

Multilayer optics for a wiggler beamline (invited)

Randall L. Headrick,^{a)} Karl W. Smolenski, and Alexander Kazimirov
Cornell High Energy Synchrotron Source, Wilson Lab, Cornell University, Ithaca, New York 14853

Chian Liu and Albert T. Macrander
Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois 60439

(Presented on 23 August 2001)

A double crystal, multilayer monochromator was designed and fabricated for a wiggler beamline at the Cornell High Energy Synchrotron Source. The monochromator consists of an internally water-cooled first substrate and a fixed-radius sagittally focusing second substrate, each coated with a multilayer consisting of 100 bilayers of tungsten/carbon with a 27 \AA d spacing. Cooled silicon substrates were fabricated with internal water cooling channels to reduce or eliminate thermal distortion. The wide energy bandpass of this multilayer along with sagittal focusing provides the best available flux for time resolved experiments. A flux 100 times that of conventional silicon monochromators is possible and allows for a finer time resolution for the crystal growth studies on this beamline. Measured reflectivities over 60% and bandwidths of 1.3%–1.8% were obtained. Results to beam currents of 350 mA show the effectiveness of the internal cooling design and have provided x-ray fluxes of 8×10^{13} photons/s/mm². © 2002 American Institute of Physics.
[DOI: 10.1063/1.1435819]

I. INTRODUCTION

Multilayers have been routinely applied as optical elements on synchrotron radiation beamlines over the past 20 years.^{1–3} Currently available commercial multilayers are suitable for a wide variety of synchrotron applications. Multilayers consist of many alternating layers of deposited thin films of low- Z and high- Z materials from which x rays can be diffracted. The ability to tailor the thickness of the layers (the d spacing) and the species of the alloys of the film allows the design of an x-ray optic optimized for a specific experimental requirement. Perfect crystals of silicon or germanium commonly used as x-ray monochromators have an intrinsic energy bandpass of roughly $10^{-4} \Delta E/E$. For many experimental applications, this narrow bandwidth is not required and a broader energy bandpass would provide increased fluxes at samples for more rapid data collection.⁴ Multilayers have an intrinsic bandpass on the order of 10^{-2} , and therefore, can provide fluxes approximately 100 times that of perfect crystals. A number of surface growth experiments at the Cornell High Energy Synchrotron Source (CHESS) A2 wiggler station are based on time resolved specular and off-specular diffuse scattering and thus a finer time resolution would be possible with increased flux.^{5,6}

Several groups have investigated the ability of multilayers to survive intense synchrotron x-ray beams all with promising results at the power levels explored.^{7–11} Of these, the work on the European Synchrotron Radiation Facility microfocus beamline has the highest specific power with 200 W absorbed by the first multilayer and the work on National Synchrotron Light Source beamline $\times 25$, using a toroidal focusing mirror before the multilayer, has the highest surface

power density at 7.6 W/mm^2 . In all of these cases, the cooling of the multilayer substrates was accomplished through contact with a cooled copper mount, often with enhanced thermal contact using a liquid metal layer.

The A2 wiggler station at the CHESS receives half the beam from a 24 pole permanent magnet wiggler installed in the Cornell Electron Storage Ring. Present operation of the ring at 350 mA provides a beam of 9450 W to A2 after most of the low energy spectrum is removed by a graphite filter and beryllium windows. A cooled flat, vertically focusing mirror absorbs roughly half the incident power. 4600 W total are incident on the first-multilayer substrate in the double crystal monochromator. The average power density incident on the first-multilayer surface is roughly 1.5 W/mm^2 , considering a typical inclination of 2.0° .

Several groups have investigated the use of curved substrates coated with multilayer films for the focusing of x rays, primarily using the Kirkpatrick–Baez (K–B) mirror geometry to generate microprobe beams.^{12,3,13} Use of the K–B geometry with wide wiggler or bend magnet beams is difficult since the length of the mirror focusing in the horizontal would be unpractical to collect the entire beam. While proposed nearly 20 years ago,^{2,14} the use of sagittally focusing multilayers does not appear to have come into widespread use. The benefits of sagittally focusing a wide beam is clear since the intensity of the full beam can be concentrated in a small spot. To capitalize on the possible flux gains in both bandpass, roughly 100 times that of silicon(111), and focusing, perhaps ten times that of an unfocused beam, a matched set of multilayers with sagittal focusing has been designed and tested for the CHESS A2 wiggler station.

^{a)}Present address: Department of Physics, University of Vermont, Burlington, VT 05401; electronic mail: rheadrick@uvm.edu

II. PAST EXPERIENCE

Initially, commercially available multilayers (5 mm \times 25 mm \times 50 mm) from Osmic, Inc.¹⁵ (specifications listed in Table III) were installed on the double crystal A2 monochromator. These silicon substrates were nearly identical to the substrates of Berman *et al.* (Ref. 11) and were indirectly cooled through contact with a water-cooled copper mount, using GaIn eutectic to aid in thermal contact. The thermal distortions caused by the intense wiggler beam led to severe blow up of the focal spot size. Only when the width of the beam on the multilayer substrate was reduced to 2 mm using cooled slits, and thus the power reduced to 700 W, could the multilayer be used without degradation of the focal spot. These distortions were purely thermal and thus reversible and in all other regards the performance of the Osmic multilayers was excellent without any degradation¹⁶ or reflectivity or bandwidth over a 4 month period of use. Based on this experience, we have designed and tested a cooled multilayer monochromator fabricated with internal water cooling channels to reduce or eliminate thermal distortion.

The A2 station receives about 2 mR of x rays from the 24-pole wiggler. At the position of the A2 station, this corresponds to a beam width of 50 mm. Since typical experiments at this station accept only about 1 mm of x rays in the horizontal direction, a large gain in the useful flux delivered to the experiment is obtained by employing horizontal focusing. The layout of the beamline with a 2.5:1 ratio of source-to-monochromator and monochromator-to-sample has proven to be nearly ideal for sagittal focusing based on silicon single-crystal monochromators. Therefore, we have designed and tested a fixed-focus cylindrical multilayer to sagittally focus x rays in the horizontal direction.

III. DESIGN AND FABRICATION

The experimental program of A2 includes numerous time-resolved measurements of growing semiconductor films, and an energy bandwidth of 1%, with the highest possible reflectivity, allows for better time resolution and faster data collection due to fluxes roughly 100 times greater than narrow bandwidth silicon crystal monochromators. New cooled substrates were designed following our past work constructing internally water-cooled silicon monochromators.¹⁷ Aside from the increased length of the substrate required to collect the entire beam reflected from an upstream mirror, no significant changes were made to the cooling design. 20 channels 1 mm wide, 3.5 mm deep, with a 1 mm fin between each of the channels pass water roughly 1 mm below the face of the substrate. Immediately upstream of the multilayer is a 600 mm long Rhodium coated water-cooled white beam mirror, set for a cutoff energy of 20 keV, which accepts the central 2 mm vertically of the wiggler beam. Substrates 170 mm \times 50 mm \times 20 mm accept the entire width of the beam, 20 mm, and can accept the 2 mm vertical height reflected from the mirror at substrate angles as low as 0.75°.

The multilayer substrates (Figs. 1 and 2) were manufactured at CHESS from Czochralski single crystal silicon due to high polishability and good thermal performance of sili-

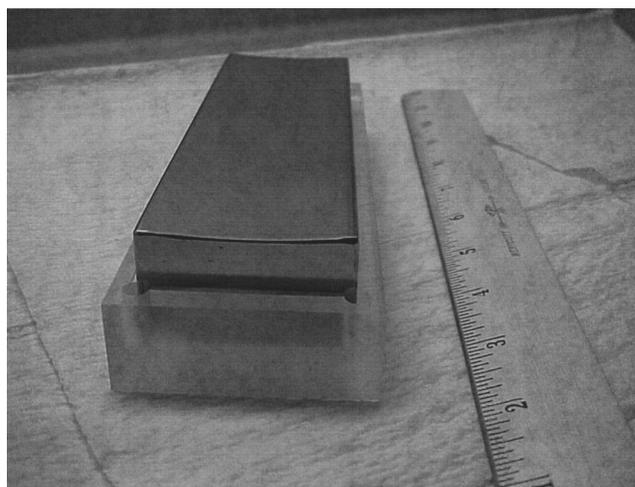


FIG. 1. Cylindrically figured second crystal, used for sagittal focusing. The silicon substrate is 150 mm \times 50 mm \times 20 mm thick. The scale on the ruler in the photograph is in inches.

con. The polished substrate surface was $\langle 110 \rangle$ oriented. In order to encapsulate the cooling channels, the substrate consists of two halves: the face plate with cooling channels and a water manifold. The mating surfaces are syton polished before any machining to ensure flat surfaces for the subsequent bonding procedure. The cooling channels were plunge cut with a diamond saw and the water manifold was machined with diamond endmills and core drills.

After machining, the two halves are bonded using a silver brazing technique.¹⁸ The silver braze imposes significant strains on the silicon due to differential thermal expansion at the bond. The effectiveness of crystal monochromators is reduced by this imposed strain, but it does not effect the multilayer performance since the substrate is polished after bonding and the diffraction occurs in the evaporated layers. A slight improvement to the bond quality was made by reverse sputter etching the mating surfaces and then depositing a protective 1000 Å layer of silver before brazing. This ensures that silver is in direct contact with the silicon, without any growth of a SiO₂ layer to disrupt the even wetting of the

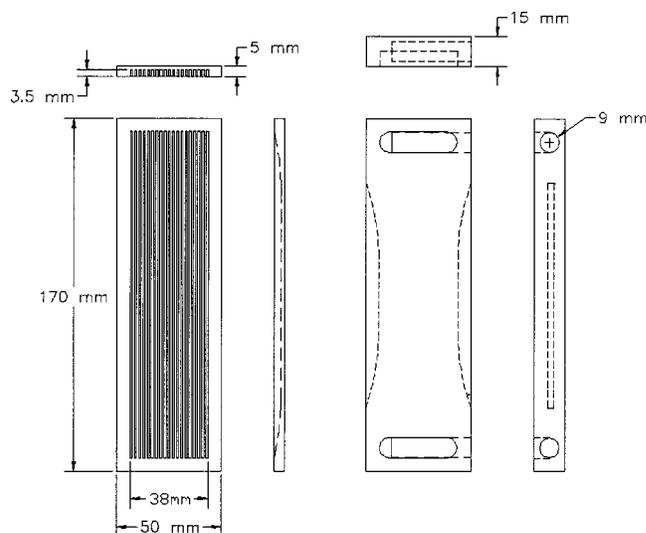


FIG. 2. Multilayer substrate.

TABLE I. Results of substrate polishing.

Substrate No.	rms roughness (Å)	Peak slope error (s)
Flat ML No. 1 (cooled)	2.0	4.9
Flat ML No. 2 (cooled)	2.5	1.2
Flat ML No. 3 (uncooled)	0.5	0.6
Flat ML No. 4 (uncooled)	0.5	0.6
Cylindrical ML	2.6	5.4

surfaces. A 25 μm foil of silver was used to braze the two surfaces together.

The flat substrates were polished by General Optics¹⁹ to specifications of 3 Å root-mean-square (rms) surface roughness and 1 s slope error with the measured results given in Table I. Uncooled substrates were easily polished to better than our specifications. Both cooled substrates, with channels 1 mm below the polished surface, exhibit poor roughness and slope errors when compared with the uncooled substrates, but still acceptable and state of the art. Substrate ML No. 1 exhibits a defect in one corner, perhaps a delamination of the bond near a fin, and thus its peak slope error does not reflect the quality of the majority of the surface. Future substrates will be made with a thicker hot wall between the polished surface and channels, slightly compromising the thermal performance, but allowing for improved surface figure errors.

The cylindrical substrate was fabricated by SESO.²⁰ It exhibited degraded roughness and figure compared to the flat uncooled test substrates, but is well-matched to the water-cooled substrates. Table I illustrates the effect of the various substrate geometries on the polishing results.

Since the multilayers operate in a high vacuum chamber, a robust connection must be made between the water supply and substrate. Copper tubing is soldered to the substrates at 65 °C using Wood's metal,²¹ a low melting point bismuth-lead-tin-cadmium alloy. Proper metalization of the silicon surface is required for consistent wetting and consists of a 500 Å layer of chromium with a final 3000 Å of gold. Ultrasonic agitation of the molten solder aids in obtaining an ultrahigh vacuum compatible joint. Routinely, the solder joint has a helium leak rate of better than 1×10^{-9} mbar-1/s. Chilled, demineralized water is fed to the substrates at a flow rate of 0.06 l/s.

Figure 3 shows the optical geometry of the sagittally focusing second substrate. Equation (1) gives the required focal length of the optic for a specific source to image geometry. From Eq. (2), it is clear that the focal length varies with wavelength:

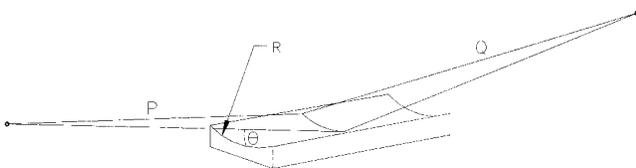


FIG. 3. Optical layout of the sagittal optic. P is the distance from source to optic, Q is the distance from optic to image.

TABLE II. Optimum energy for sagittal focusing at CHESS stations with a 225 mm fixed-curvature cylindrical multilayer.

Beamline	Optimum energy (keV)
F3	8.1
A2 (24 pole)	9.98
A2 (48 pole)	11.08

$$\frac{1}{f_s} = \frac{1}{P} + \frac{1}{Q}, \quad (1)$$

$$f_s = R_s/2 \sin \theta = R_s d/\lambda, \quad (2)$$

and that in order to tune over a range of energies, the sagittal radius of the multilayer needs to vary with the Bragg angle, or conversely, the optimal focal spot location moves with wavelength. For this prototype optic, an adjustable radius was rejected due to its complexity and due to space constraints the diffractometer/surface chamber location cannot be readily moved, forcing the selection of a fixed radius optimal only for a single energy. A radius of 225 mm was selected corresponding to an energy of 10 keV at the A2 wiggler station. Table II shows the energy for optimum focus for a multilayer of $d=27.0$ Å considering the focusing geometry of the CHESS F3 (bend magnet test station) and A2 wiggler beamlines. In the Fall of 2001, the A2 wiggler will be upgraded to a 48-pole device and the storage ring optics reconfigured for a reduced source size. For completeness, the predicted effects of this upgrade were included in Tables II and IV.

IV. TESTING AND RESULTS

The four multilayers produced were characterized off line before installation on A2. The substrates deposited by Osmic were tested on a conventional source (Cu- $K\alpha$) while the Advanced Photon Source (APS)-deposited substrates were tested on the CHESS F3 station with a monochromatic 10 keV beam. Table III summarizes the measured reflectivity, averaged over many points on the surface, and likewise, the average bandwidth. Performance of both sets of multilayers was remarkably similar. Both cooled substrates had poorer reflectivity and increased bandwidth, indicating the effect of the increased roughness and figure error caused by the subsurface cooling channels.

Flat ML1 and the cylindrical ML were tested as a monochromator pair at the F3 station with the energy tuned to the optimum energy for focusing near the center of the F3 station (8.1 keV). The profile of the focal spot was measured by rastering a 200 μm pinhole across the entire beam and mea-

TABLE III. Summary of multilayers produced and tested.

Multilayer sample	Coating	Average reflectivity	Average bandwidth
Flat ML 1 (cooled)—APS	18 Å C/9 Å W	62%	2.5%
Flat ML 2 (cooled)—Osmic	15 Å B ₄ C/7.5 Å W	62%	2.5%
Flat 3 (uncooled)—Osmic	15 Å B ₄ C/7.5 Å W	74%	1.3%
Flat ML 4 (uncooled)—APS	18 Å C/9 Å W	74%	1.8%
Cylindrical ML—APS	18 Å C/9 Å W	45%	1.4%

TABLE IV. Focal spot size.

Beamline	p (m)	q (m)	Calculated (mm)	Measured (mm)
F3	16.6	4.6	1.66	1.75
A2 (23 pole)	19.5	6.6	2.23	2.83
A2 (48 pole)	30.8	6.6	0.71	...

suring the transmitted flux in an ion chamber. Translating the pinhole along the beam direction and repeating the scan allowed the best focal spot to be found. The positron beam source size for the F3 station is 6 mm horizontal \times 1 mm vertical, which is focused in a 3.6:1 geometry to a full width at half maximum of 1.75 mm, just slightly greater than the expected 1.66 mm.

Subsequent to these tests, the monochromator pair was installed at the CHESS A2 stations where they were employed for CHESS user experiments requiring a high-flux monochromatic x-ray beam. An x-ray flux at the experiment of 8×10^{13} photons/s/mm² was routinely obtained. This figure can be compared to the theoretical estimate of the flux of $\sim 10^{15}$ photons/s, taking into account the source flux, measured reflectivity of the optics, and absorption by windows.²² There is a small discrepancy between measured and experimental values that is likely to be a result of reduced reflectivity at the edges of the cylindrical multilayer; this effect was noted during testing, but has not yet been characterized in detail. Table IV shows a comparison of the focusing properties at the F3 and A2 stations, including the expected focal spot size for the station after the wiggler upgrade.

ACKNOWLEDGMENTS

This work is based upon research conducted at CHESS, which is supported by the National Science Foundation, under Award No. DMR-9311772. The authors sincerely thank the staff of the APS and Jim Wood of Osmic Inc. for their assistance in testing and preparing the multilayers. Technical assistance in the preparation of the substrates was provided by Ron Kemp, Walter Protas, and Gerhard Schmidt of Cornell University.

- ¹R. Day, J. Grosso, R. Bartlett, and T. Barbee, Nucl. Instrum. Methods Phys. Res. **208**, 245 (1983).
- ²D. H. Bilderback, B. M. Lairson, T. W. Barbee, G. E. Ice, and C. J. Sparks, Nucl. Instrum. Methods Phys. Res. **208**, 251 (1983).
- ³J. H. Underwood, A. C. Thompson, Y. Wu, and R. D. Giauque, Nucl. Instrum. Methods Phys. Res. A **266**, 296 (1988).
- ⁴G. B. Stephenson, Nucl. Instrum. Methods Phys. Res. A **266**, 447 (1988).
- ⁵R. L. Headrick, S. Kycia, A. R. Woll, J. D. Brock, and R. Murty, Phys. Rev. B **58**, 4818 (1998).
- ⁶R. Murty, T. Curcic, A. Judy, B. H. Cooper, A. R. Woll, J. D. Brock, S. Kycia, and R. L. Headrick, Phys. Rev. Lett. **80**, 4713 (1998).
- ⁷E. Ziegler, Y. Lepptre, S. Joksch, V. Saile, S. Mourikis, P. J. Viccaro, G. Rolland, and F. Laugier, Rev. Sci. Instrum. **60**, 1999 (1989).
- ⁸J. B. Kortright, S. Joksch, and E. Ziegler, J. Appl. Phys. **69**, 168 (1991).
- ⁹E. Ziegler, G. Marot, A. K. Freund, S. Joksch, H. Kawata, L. E. Berman, and M. Iarocci, Rev. Sci. Instrum. **63**, 496 (1992).
- ¹⁰P. Deschamps, P. Engstrom, S. Fiedler, C. Riekel, S. Wakatsuki, P. Highij, and E. Ziegler, J. Synchrotron Radiat. **2**, 124 (1995).
- ¹¹L. E. Berman, Z. Yin, S. B. Dierker, E. Dufresne, S. Mochrie, O. Tsui, S. K. Burley, F. Shu, X. Xie, M. S. Capel, and R. M. Sweet, *Proceedings of the 10th U.S. National Conference, Synchrotron Radiation Instrumentation*, edited by Ernest Fontes, pp. 71–79.
- ¹²D. H. Bilderback, B. M. Lairson, T. W. Barbee, Jr., G. E. Ice, and C. J. Sparks, Jr., Nucl. Instrum. Methods Phys. Res. **208**, 251 (1983).
- ¹³A. Bakulin, S. M. Durbin, C. Liu, J. Erdmann, A. T. Macrander, and J. Jach, Proc. SPIE **3448**, 218 (1998).
- ¹⁴G. E. Ice and C. J. Sparks, Jr., Nucl. Instrum. Methods Phys. Res. **222**, 121 (1984).
- ¹⁵General Optics, Inc., 554 Flinn Avenue, Moorpark, CA 93021.
- ¹⁶A carbon film which built up on the surface of the first multilayer was easily removed using hydrogen peroxide; this was most likely due to operation of the monochromator vacuum chamber at a relatively poor 1×10^{-5} Torr, compared with common practice for mirror vacuum chambers of 1×10^{-8} Torr or better.
- ¹⁷K. W. Smolenski, Q. Shen, and P. Doing, Proc. SPIE **3151**, 181 (1997).
- ¹⁸K. W. Smolenski, C. Conolly, P. Doing, B. Kiang, and Q. Shen, Proc. SPIE **2855**, 220 (1996).
- ¹⁹General Optics, Inc., 554 Flinn Avenue, Moorpark, CA 93021.
- ²⁰SESO (Société Européenne de Systèmes Optiques), Pôle d'Activités d'Aix-les-Milles, 305 rue Louis Armand, 13792-Aix en Provence cédex 3, France.
- ²¹A. K. Freund, J. R. Arthur, and L. Zhang, Proc. SPIE **3151**, 216 (1997).
- ²²The flux at the experiment is measured using a He-filled ion chamber, and a 1 mm by 1 mm slit. The total flux can be estimated by multiplying this value (expressed in units of photons/s/mm²) by the focal spot width and the vertical acceptance of the optics (3 mm). By this estimate, the measured flux at the A2 station converts to $\sim 7 \times 10^{14}$ photons/s, slightly below the theoretical estimate.